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Database:	US Pre-Grant Publication Full-Text Database US Patents Full-Text Database US OCR Full-Text Database EPO Abstracts Database JPO Abstracts Database Derwent World Patents Index IBM Technical Disclosure Bulletins
Term:	L11 and extrusion
Display:	10 Documents in Display Format: CIT Starting with Number 1
Generate: <input type="radio"/> Hit List <input checked="" type="radio"/> Hit Count <input type="radio"/> Side by Side <input type="radio"/> Image	

Search History

DATE: Thursday, April 29, 2004 [Printable Copy](#) [Create Case](#)

Set Name Query

side by side

DB=USPT; PLUR=YES; OP=ADJ

Set Name	Hit Count	Set Name
<u>L12</u> L11 and extrusion	49	<u>L12</u>
<u>L11</u> L10 and microporous and membrane	75	<u>L11</u>
<u>L10</u> polysulfone and solvent and heating and quench	273	<u>L10</u>
<u>L9</u> 6218441.pn.	1	<u>L9</u>
<u>L8</u> L7 and microfiltration	4	<u>L8</u>
<u>L7</u> melt-extrusion and membrane and polysulfone	22	<u>L7</u>
<u>L6</u> polysulfone embrane and melt same extrusion	0	<u>L6</u>
<u>L5</u> L4 and micropores	2	<u>L5</u>
<u>L4</u> l2 and melt same extrusion	11	<u>L4</u>
<u>L3</u> L2 and melt and temperature and heat?	1	<u>L3</u>
<u>L2</u> L1 and polysulfone same membrane	257	<u>L2</u>
<u>L1</u> 210/500.41.ccls.	356	<u>L1</u>

END OF SEARCH HISTORY

First Hit Fwd Refs Generate Collection

L5: Entry 1 of 2

File: USPT

Feb 7, 1989

DOCUMENT-IDENTIFIER: US 4802942 A

TITLE: Method of making multilayer composite hollow fibers

Brief Summary Text (23):

In the practice of the present invention, a variety of polymers may be used as the polymer (A') for forming the nonporous separating membrane layer (A). Examples of such polymers include silicones, polyurethanes, cellulosics, polyolefins, polysulfones, polyvinyl alcohol, polyesters, polyethers, polyamides and polyimides.

Brief Summary Text (35):

For this purpose, it is preferable to employ an extrusion temperature ranging from the melting point of the polymer (B') to a temperature about 80.degree. C. higher than the melting point, and it is also preferable to employ a spinning draw ratio of not less than 30. If the extrusion temperature is higher than the melting point by more than about 80.degree. C., it is difficult to achieve stable spinning. If the spinning draw ratio is less than 30, the melt-spun polymer (B') has a low degree of orientation and cannot be satisfactorily drawn in a subsequent stretching step. As a result, it is difficult to form micropores in the layers (B).

Brief Summary Text (37):

Where the polymer (A') forming the layer (A) is a noncrystalline polymer or a polymer containing a solvent or a plasticizer, the above-described stretching process does not make the layer (A) porous, but allows it to be amenably stretched with a gradual reduction in thickness. If the polymer (A') forming the layer (A) has a lower melting point than the polymer (B'), the extrusion temperature should be within the aforesaid extrusion temperature range but above a temperature 60.degree. C. higher than the melting point of the polymer (A'), or the first-stage stretching temperature should be within the aforesaid stretching temperature range but above a temperature 70.degree. C. lower than the melting point of the polymer (A'). If the polymer (A') forming the layer (A) is of the same type as the polymer (B') but has a melt index different from that of the polymer (B'), it is preferable to reduce its melt viscosity and thereby decrease the stress applied to the polymer melt for the purpose of suppressing the orientation and crystallization of the polymer (A'). More specifically, the layers (B) alone can be made porous by employing an extrusion temperature above a temperature 30.degree. C. higher than the melting point of the polymer (A').

Detailed Description Text (3):

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, polyethylene having a density of 0.968 g/cm.³ and a melt index of 5.5 was melt-extruded through the innermost and outermost orifices of the nozzle, while polyethylene having a density of 0.920 g/cm.³ and a melt index of 5.0 was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 160.degree. C. and an extrusion line speed of 5 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 800 m/min.

Detailed Description Text (8):

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e ge

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, polypropylene having a density of 0.913 g/cm.^{sup.3} and a melt index of 15 was melt-extruded through the innermost and outermost orifices of the nozzle, while poly-4-methylpentene-1 having a density of 0.835 g/cm.^{sup.3} and a melt index of 20 was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 250.degree. C. and an extrusion line speed of 5 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 400 m/min.

Detailed Description Text (13):

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, the same polypropylene as used in Example 2 was melt-extruded through the innermost and outermost orifices of the nozzle, while ethyl cellulose having a degree of ethoxylation of 49% was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 205.degree. C. and an extrusion line speed of 4 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 300 m/min.

Detailed Description Text (18):

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, polyethylene having a density of 0.965 g/cm.^{sup.3} and a melt index of 5.2 was melt-extruded through the innermost and outermost orifices of the nozzle, while an ultraviolet-curable silicone resin (commercially available from Toshiba Silicone Co., Ltd., under the trade name of TUV6020) was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 160.degree. C. and an extrusion line speed of 10 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 350 m/min.

Detailed Description Text (24):

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, the same polyethylene as used in Example 1 was melt-extruded through the innermost and outermost orifices of the nozzle, while a mixture of acetylcellulose having a degree of acetylation of 40% and polyethylene glycol used as a plasticizer (in an amount of 50% by weight based on the acetylcellulose) was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 170.degree. C. and an extrusion line speed of 7.5 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 300 m/min.

Detailed Description Text (29):

A hollow fiber was melt-spun from a combination of two different materials by using a spinning nozzle having three concentrically arranged annular orifices. Specifically, the same polypropylene as used in Example 2 was melt-extruded through the innermost and outermost orifices of the nozzle, while a mixture of polyvinyl alcohol having a degree of saponification of 99 mole % and a degree of polymerization of 1700 and glycerol used as a plasticizer (in an amount of 50% by weight based on the polyvinyl alcohol) was melt-extruded through the intermediate orifice of the nozzle. This spinning was carried out at an extrusion temperature of 200.degree. C. and an extrusion line speed of 7 cm/min., and the hollow fiber so formed was taken up at a take-up speed of 300 m/min.

Current US Cross Reference Classification (7):

210/500.41

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Hit List

Search Results - Record(s) 1 through 4 of 4 returned.

1. Document ID: US 6218441 B1

L8: Entry 1 of 4

File: USPT

Apr 17, 2001

US-PAT-NO: 6218441

DOCUMENT-IDENTIFIER: US 6218441 B1

** See image for Certificate of Correction **

TITLE: Melt-spun polysulfone semipermeable membranes and methods for making the same

DATE-ISSUED: April 17, 2001

INVENTOR-INFORMATION:

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US-CL-CURRENT: 264/129; 210/500.21, 210/500.41, 264/143, 264/148, 264/177.14,
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528/491, 528/502D, 528/503

Full Title Citation Front Review Classification Date Reference Claims KWMC Drawn As

2. Document ID: US 5888434 A

L8: Entry 2 of 4

File: USPT

Mar 30, 1999

US-PAT-NO: 5888434

DOCUMENT-IDENTIFIER: US 5888434 A

TITLE: Process for making a microporous membrane from a blend containing a poly(phenylene sulfide) polymer, an amorphous polymer, and optionally a solvent

DATE-ISSUED: March 30, 1999

INVENTOR-INFORMATION:

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US-CL-CURRENT: 264/28, 264/184, 264/203, 264/205, 264/210.3, 264/210.4, 264/210.6,
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264/211.2, 264/216, 264/235, 264/235.6, 264/288.8, 264/289.6, 264/290.5, 264/346,
264/41

Full | Title | Citation | Front | Review | Classification | Date | Reference | Drawings | Claims | KDDC | Drawn Obj

 3. Document ID: US 5275725 A

L8: Entry 3 of 4

File: USPT

Jan 4, 1994

US-PAT-NO: 5275725

DOCUMENT-IDENTIFIER: US 5275725 A

TITLE: Flat separation membrane leaf and rotary separation apparatus containing flat membranes

DATE-ISSUED: January 4, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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US-CL-CURRENT: 210/321.67, 210/321.75, 210/321.84, 210/331, 210/500.21, 210/506

Full | Title | Citation | Front | Review | Classification | Date | Reference | Drawings | Claims | KDDC | Drawn Obj

 4. Document ID: US 5205968 A

L8: Entry 4 of 4

File: USPT

Apr 27, 1993

US-PAT-NO: 5205968

DOCUMENT-IDENTIFIER: US 5205968 A

TITLE: Process for making a microporous membrane from a blend containing a poly(etheretherketone)-type polymer, an amorphous polymer, and a solvent

DATE-ISSUED: April 27, 1993

INVENTOR-INFORMATION:

h e b b g e e e f e c ef b e

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US-CL-CURRENT: 264/28, 210/500.27, 210/500.28, 210/500.41, 264/184, 264/203,
264/210.3, 264/210.4, 264/210.6, 264/211.18, 264/211.19, 264/211.2, 264/216,
264/235, 264/235.6, 264/346, 264/41

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Terms	Documents
L7 and microfiltration	4

Display Format: [CIT](#) | [Change Format](#)

[Previous Page](#) [Next Page](#) [Go to Doc#](#)

First Hit Fwd Refs [Generate Collection](#) [Print](#)

L8: Entry 1 of 4

File: USPT

Apr 17, 2001

DOCUMENT-IDENTIFIER: US 6218441 B1

** See image for Certificate of Correction **TITLE: Melt-spun polysulfone semipermeable membranes and methods for making the sameAbstract Text (1):

The present invention discloses, inter alia, a composition useful for producing a homogeneous, semipermeable membrane, the composition comprising (1) a polysulfone compound, (2) a solvent, such as sulfolane, antipyrine, δ -valerolactam, diethyl phthalate, and mixtures thereof, and (3) a non-solvent, such as poly(ethylene glycol), di(ethylene glycol), tri(ethylene glycol), glycerol, and mixtures thereof. Another aspect of this invention discloses methods for fabricating semipermeable membranes by homogeneously mixing the composition of the polysulfone compound, solvent, and non-solvent, melting the composition, and melt-spinning the molten composition. Another aspect of the present invention includes homogeneous, melt-spun, semipermeable membranes useful for liquid separation processes, such as, but not limited to, microfiltration, ultrafiltration, dialysis, and reverse osmosis.

Brief Summary Text (2):

The present invention concerns polysulfone semipermeable membranes and methods for making the same. More particularly, the invention pertains to melt-spun polysulfone semipermeable membranes.

Brief Summary Text (4):

Contemporary semipermeable membranes are available in a variety of forms such as sheets, tubes, and hollow fibers. A "hollow fiber" is generally a hollow cylindrical structure in which the wall functions as a permeable, non-permeable, or semipermeable (i.e., selectively permeable) membrane depending upon the application. Generally, hollow fibers are used as cylindrical membranes that permit selective exchange of materials across the walls.

Brief Summary Text (5):

Liquid-separation processes utilizing membranes having selective permeabilities, such processes including ultrafiltration, microfiltration, dialysis, reverse osmosis, or the like, require a variety of materials adapted for diversified applications. For example, semipermeable membranes are currently favored for use in extracorporeal blood treatments including hemodialysis, hemofiltration, and hemodiafiltration. In such cases, the membranes typically comprise hollow fibers bundled together and assembled in a casing in a manner allowing blood to flow simultaneously in a parallel manner through the lumina of the fibers while a blood-cleansing liquid is simultaneously passed through the casing so as to bathe the exterior surfaces of the hollow fibers with the liquid.

Brief Summary Text (6):

Compounds utilized for selectively permeable membranes have included polymers, such as cellulose, cellulose acetate, polyamide, polyacrylonitrile, polyvinylalcohol, polymethyl methacrylate, polysulfone, polyolefin, or the like, depending upon the use of the membranes. Polysulfone compounds are of particular interest as they have, inter alia, excellent physical and chemical properties, such as resistance to

heat, resistance to acids, resistance to alkali, and resistance to oxidation. Polysulfone compounds have been found to be biocompatible, capable of forming excellent pores and interstitia, and chemically inert to such compounds as bleach, disinfectants, and salt solutions. Polysulfone compounds can be sterilized by a number of methods, such as ethylene oxide (EtO), gamma irradiation, steam autoclave, and heated citric acid. Additionally, polysulfone compounds possess sufficient strength and resistance to wear to withstand repeated use and sterilization cycles.

Brief Summary Text (7):

Conventionally, polysulfone hollow fibers have been formed by solution-spinning techniques. Producing polysulfone hollow fibers by solution-spinning techniques typically involves dissolving a polysulfone compound in a relatively large amount of an aprotic solvent and a non-solvent, then extruding the solution through a spinneret. For solution spinning, a "solvent" is a compound in which the polysulfone compound substantially dissolves at the membrane-fabrication temperature (i.e., ambient temperature). For solution spinning, a "non-solvent" is a compound in which the polysulfone compound is substantially insoluble at the membrane-fabrication temperature. For solution-spinning techniques, the solvents must be sufficient to substantially dissolve the polysulfone compound and produce a homogeneous liquid at ambient temperature (membrane fabrication temperature).

Brief Summary Text (8):

The solvents and non-solvents utilized for solution-spinning techniques require that the membranes be extensively leached and rinsed after fabrication, as even residual amounts left in the membranes can cause unacceptable contamination of fluids treated using the membranes. Avoiding such contamination is particularly important in membranes used for the treatment of blood by dialysis or the desalination of water by reverse osmosis. When fabricating hollow-fiber membranes utilizing solution spinning techniques, removal of the core liquid used to form the fiber lumen is especially difficult. Following removal of the solvents, non-solvents, and core liquid, a non-volatile, water-soluble compound must then be added to preserve the membrane pore structure prior to drying the membrane. The non-volatile material also serves as a surfactant for later rewetting of the membranes. Such a process is known as "replasticization."

Brief Summary Text (9):

Solution-spinning techniques require the inclusion of large amounts of solvents and non-solvents many of which are generally toxic and can be difficult to extract from the resulting polysulfone fiber. Moreover, the significant amount and high level of toxicity of certain solvents and non-solvents removed from the fibers may create a hazardous waste-disposal problem.

Brief Summary Text (10):

Moreover, conventional solution-spinning techniques produce asymmetric polysulfone membranes (i.e., non-homogeneous membrane porosity progressing through the thickness dimension of the membrane). That is, a non-homogeneous membrane has a dense skin or micro-porous barrier layer on one (or both) of the major surfaces of the membrane. The dense skin or micro-porous barrier layer comprises a relatively small portion of the membrane but contributes a disproportionately large amount of control on the permeability characteristics of the membrane.

Brief Summary Text (11):

Accordingly, there is a need for a polysulfone composition and simple method for the production of polysulfone semipermeable membranes which composition and method minimizes toxic waste by-products. Additionally, there is a need for a method for the production of polysulfone semipermeable membranes wherein the solvents, non-solvents, and processing aids used in the manufacture of the membranes are easily removed from the membranes after fabrication and/or are of relatively low toxicity. There is also a need for polysulfone semipermeable membranes having a more uniform

structure throughout the thickness dimension (i.e., a homogeneous polysulfone membrane) so that the entire thickness dimension controls the permeability of the membrane.

Brief Summary Text (13):

In general, the present invention provides, inter alia, a novel method and polysulfone composition for preparing a homogeneous, semipermeable polysulfone membrane by melt-spinning. The polysulfone composition comprises a liquid mixture of a polysulfone compound, a solvent and, optionally, a non-solvent that are relatively non-toxic and that preferably do not deleteriously affect the environment.

Brief Summary Text (17):

The solvent and non-solvent are present in a ratio useful to form a semipermeable, polysulfone membrane useful for performing liquid-separation processes.

Brief Summary Text (18):

According to another aspect of the invention, a "melt-spinning" or "melt-extrusion" method is provided for producing semipermeable, polysulfone membranes. The melt-spinning method includes the steps of: (1) forming a composition comprising a polysulfone compound, a solvent selected from the foregoing group of candidate solvents and preferably selected from the group consisting of tetramethylene sulfone, antipyrine, .delta.-valerolactam, diethyl phthalate, and mixtures thereof, and, optionally, a non-solvent selected from the foregoing group of candidate non-solvents and preferably selected from the group consisting of poly(ethylene glycol), di(ethylene glycol), tri(ethylene glycol), glycerol, and mixtures thereof; (2) heating the composition to a temperature at which the composition becomes a homogeneous liquid (i.e., a temperature greater than ambient); (3) extruding the composition through an extrusion die (such as a single or multi-holed hollow-fiber die (termed a "spinneret")); and (4) passing the extrudate through a quench zone in which the extrudate gels and solidifies, thereby forming the membrane.

Brief Summary Text (19):

According to another aspect of the present invention, melt-spun, semipermeable, polysulfone membranes are provided having a uniform structure throughout the thickness dimension of the membrane (i.e., a "homogeneous" membrane structure) useful for liquid separations, such as, but not limited to, microfiltration, ultrafiltration, reverse osmosis, and dialysis.

Drawing Description Text (2):

FIG. 1 illustrates a preferred embodiment of the process for fabricating homogeneous polysulfone hollow fibers (as a representative membrane configuration) according to the present invention.

Drawing Description Text (3):

FIG. 2 illustrates an alternative process for fabricating homogeneous polysulfone hollow fibers according to the present invention.

Drawing Description Text (4):

FIG. 3 is a three-component diagram showing the proportions of polysulfone compound, solvent, and non-solvent which are combined in representative melt-spin compositions according to the invention.

Drawing Description Text (5):

FIG. 4 is a scanning electron microscope photograph of a representative homogeneous, polysulfone hollow fiber according to the present invention.

Drawing Description Text (6):

FIG. 5 is a schematic diagram of a hemodialyzer including homogeneous polysulfone hollow-fiber membranes of the present invention.

Detailed Description Text (2):

This invention encompasses, inter alia, compositions useful for forming, by melt-spinning, polysulfone semipermeable membranes. The compositions comprise a polysulfone compound, a solvent and, optionally, a non-solvent. In the composition, the solvent and optional non-solvent are present in a ratio useful to form a semipermeable membrane useful for performing liquid separation processes. Membranes that are melt-spun using such compositions are homogeneous. That is, the melt-spun membranes are symmetric such that the membranes have a substantially uniform structure throughout the thickness dimension of the membranes, as illustrated in the scanning electron microscope photograph in FIG. 4 of a hollow fiber made using such a composition. As defined herein, a "homogeneous" polysulfone membrane is a membrane in which each portion or section of the membrane contributes its substantially proportional share to the permeability characteristics of the membrane.

Detailed Description Text (3):

Polysulfone compounds and their synthesis are well-known in the art. Preferred polysulfone compounds useful in this invention satisfy the formula:

Detailed Description Text (4):

wherein R.sub.1, and R.sub.2 (which can be the same or different) are groups such as alkanes, alkenes, alkynes, aryls, alkyls, alkoxyis, aldehydes, anhydrides, esters, ethers, and mixtures thereof, each such group having fifty or fewer carbon atoms and including both straight-chained and branched-chained structures. Preferred polysulfone compounds useful in this invention have a melt flow index (MFI) in a range of from about 1.7 dg/min to about 9.0 dg/min as measured according to the American Standard Test Method (ASTM) for Flow Rates of Thermoplastics by Extrusion Plastometer, ASTM D 1238-94a. Good results have been achieved when the polysulfone compounds have a MFI of from about 2.0 dg/min to about 5.0 dg/min. Preferred polysulfone compounds useful in this invention include, but are not limited to polyarylsulfones, for example, bisphenol A polysulfone, polyether sulfone, polyphenyl sulfone, and mixtures thereof. Especially good results have been achieved utilizing bisphenol A polysulfone.

Detailed Description Text (5):

A "solvent for the polysulfone compound" is defined herein as a compound having the following characteristics: a boiling point of at least about 150.degree. C., a solvating power to dissolve from about 8 weight percent to about 80 weight percent of the polysulfone compound at a temperature in a range from about 50.degree. C. to about 300.degree. C. The solvent preferably can dissolve from about 8 weight percent to about 80 weight percent of a polyarylsulfone.

Detailed Description Text (7):

A "non-solvent for the polysulfone compound" is defined herein as a compound having the following characteristics: a boiling point of at least about 150.degree. C., a solvating power sufficiently low to dissolve less than about 5 weight percent of the polysulfone compound at a temperature in a range from about 50.degree. C. to about 300.degree. C.

Detailed Description Text (9):

The concentrations of the components in the composition may vary and are dependent upon variables many of which can be readily worked out with simple bench experiments. For example, miscibility of the composition at the melt-extrusion temperature is one factor to be considered in determining a suitable component concentration. Miscibility of polysulfone compound solutions can be readily determined empirically by methods known in the art. (Whether or not the components of a composition are miscible is readily apparent.) The end use of the membrane is another factor in determining the appropriate blend composition because the preferred pore size of the membrane and transport rate of liquids and solutes

through the membrane vary depending upon the intended fiber end use.

Detailed Description Text (10):

In the case of membranes useful for microfiltration of liquids, the concentration of the polysulfone compound is preferably at least about 8 weight percent, more preferably at least about 12 weight percent. The concentration of the solvent is preferably at least about 40 weight percent, more preferably at least about 60 weight percent. The concentration of the non-solvent, if present, is preferably at least about 1 weight percent, and more preferably at least about 5 weight percent.

Detailed Description Text (11):

In the case of membranes useful for ultrafiltration or dialysis, the concentration of the polysulfone compound is preferably at least about 18 weight percent, more preferably at least about 25 weight percent. The concentration of the solvent is preferably at least about 40 weight percent, more preferably at least about 45 weight percent. Concentration of the non-solvent, if present, is preferably at least about 1 weight percent, more preferably at least about 5 weight percent.

Detailed Description Text (12):

If the non-solvent is present, solvent to non-solvent ratios (i.e., a solvent to non-solvent ratio "sufficient to form a semipermeable membrane useful for liquid separation processes") are preferably about 0.95:1 to about 80:1, and more preferably about 2:1 to about 10:1. For example, as shown in FIG. 3, for a three-component composition (for melt-spinning polysulfone hollow fibers) comprising bisphenol A polysulfone, sulfolane (the solvent), and poly(ethylene glycol) (the non-solvent), acceptable amounts of the polysulfone compound, solvent, and non-solvent lie within the area bounded by the extremes of each component which generate the area A, B, C. Any of the specific compositions consisting of an amount of each of the three components within the area A, B, C of FIG. 3 are suitable for melt spinning into hollow-fiber membranes.

Detailed Description Text (13):

In the case of membranes useful for reverse osmosis of liquids, the concentration of polysulfone is preferably at least about 30 weight percent, more preferably at least about 35 weight percent. The concentration of the solvent is preferably at least about 12 weight percent, more preferably at least about 20 weight percent. If present, the concentration of the non-solvent is preferably at least about 1 weight percent, more preferably at least about 5 weight percent.

Detailed Description Text (14):

The compositions of this invention may be used to fabricate polysulfone semipermeable membranes useful for "liquid-separation processes." As defined herein, such processes include, but are not limited to, microfiltration, ultrafiltration, dialysis, and reverse osmosis. FIG. 5 shows a representative liquid-separation device configured for use as an extracorporeal blood treatment device, specifically a hemodialyzer. The hemodialyzer 10 comprises an outer casing 12, end caps 14, a dialysate inlet 16, a dialysate outlet 18, a blood inlet 20, a blood outlet 22, and a bundle of fibers 24 potted in the outer casing. The outer casing defines a dialysate compartment, and the lumina of the fibers form a blood compartment. As blood flows through the lumina of the fibers in a parallel fashion, dialysate flows counter-currently through the dialysate compartment.

Detailed Description Text (15):

Membranes of the present invention may be fabricated by alternative method schemes as illustrated in FIGS. 1 and 2. A number of method schemes may be followed depending upon the optional method steps chosen to develop the desired polysulfone membrane.

Detailed Description Text (16):

In one preferred method according to the present invention, a polysulfone

composition of polysulfone compound, solvent, and optional non-solvent is precompounded in a high-shear mixer, melted, extruded (as hollow fibers), quenched (FIG. 1), and then wound on cores or reels using any number of commercially available winders, such as Leesona winders. In such a method, adequate care should be taken to maintain a slight tension on the hollow fibers during winding. In another preferred method according to the present invention, the polysulfone composition is precompounded in a high-shear mixer, melted, extruded through a strand die (to form solid strands), cooled, pelletized, remelted, extruded (to form hollow fibers), quenched, and then wound (FIG. 2). In still another preferred method according to the present invention, the polysulfone composition is precompounded, melted, extruded (as hollow fibers), quenched, wound, held dry for a period of time, soaked in a liquid that is substantially a non-solvent for the polysulfone compound and stored in the soaking liquid for up to 15 days (FIG. 1). In yet another preferred method according to the present invention, a polysulfone composition is precompounded in a high-shear mixer, melted, extruded (as hollow fibers), quenched, wound, soaked, leached, rinsed, replasticized, and then dried in an oven (preferably a convection oven) (FIG. 1). In another preferred method according to the present invention, the polysulfone composition is precompounded, melted, extruded (as solid strands), cooled, pelletized, remelted, extruded (as hollow fibers), quenched, wound, and then held dry in air followed by soaking in a liquid that is substantially a non-solvent for the polysulfone compound (FIG. 2). In yet another preferred method according to the present invention, a polysulfone composition is precompounded, melted, extruded (as solid strands), cooled, pelletized, remelted, extruded (as hollow fibers), quenched, wound, soaked, leached, rinsed, replasticized, and dried (FIG. 2).

Detailed Description Text (17):

The components of the composition (i.e., the polysulfone compound, solvent, and optional non-solvent) to be extruded are combined and homogenized prior to extrusion by mixing in a convenient manner with conventional mixing equipment, as for example, a high-shear mixer, such as a compounding twin-screw extruder. The components of the extrusion composition may also be combined and homogenized directly in a meltpot provided with suitable agitation of the molten liquid. Alternatively, a polysulfone extrusion composition may be homogenized by extruding a molten composition through a strand die, cooling the global extrudate, and grinding or pelletizing the extrudate to a particle size readily-fed to a heated, single-screw or twin-screw extruder. Alternatively, other heating/homogenizing methods known to those skilled in the art may be utilized to produce a homogeneous, molten liquid for extrusion (termed a "melt").

Detailed Description Text (18):

The melt is heated to a temperature that facilitates preparation of a homogeneous liquid possessing a viscosity suitable for extrusion. The temperature should not be so high as to cause significant degradation of the polysulfone, the solvent, or the non-solvent. The temperature should not be so low as to render the liquid too viscous for extrusion. For example, when the melt comprises bisphenol A polysulfone, the extrusion temperature is preferably at least about 50.degree. C., more preferably at least about 75.degree. C. The extrusion temperature is preferably less than about 300.degree. C., more preferably less than about 220.degree. C.

Detailed Description Text (19):

The viscosity of the melt should not be so high as to be too viscous to be extruded at temperatures that do not deleteriously affect the polysulfone compound. The viscosity, however, of the melt must not be so low that the extrudate cannot maintain a desired shape upon exiting the extrusion die. The melt may be extruded in a variety of shapes such as, but not limited to, hollow-fibers, tubes, sheets, and hollow-fibers with fins. The extrudate may be aided in retaining its desired shape upon extrusion by cooling.

Detailed Description Text (20):

For making hollow-fiber membranes, the melt is extruded through a hollow-fiber die (spinneret). The spinneret typically is multi-holed and, thus, produces a tow of multiple hollow fibers. The spinneret typically includes a means for supplying a fluid (gas or liquid) to the core or "lumen" of the extrudate. The core fluid is used to prevent collapse of the hollow fibers as they exit the spinneret. The core fluid may be a gas, such as nitrogen, air, carbon dioxide, or other inert gas, or a liquid which is a non-solvent for the polysulfone compound, such as, but not limited to, water, poly(ethylene glycol), di(ethylene glycol), tri(ethylene glycol), glycerol, and mixtures thereof. Mixtures of solvents and non-solvents may be used as long as the mixture is not a solvent for the polysulfone compound. Alternatively, the melt may first be extruded as solid strands through a single or multi-holed strand die and the resulting solid strands cooled and pelletized to a particle size readily fed to a single-screw or twin-screw extruder (FIG. 2). In this alternative method of production, the particles are remelted and then extruded through a single-holed or multi-holed spinneret to form hollow fibers, as described above.

Detailed Description Text (21):

The extrudate exiting extrusion die enters one or more quench zones. The environment of a quench zone may be gaseous or liquid. Within the quench zone, the extrudate is subjected to sufficient cooling to cause gelation and solidification of the membrane. In an embodiment of the method of the present invention, the time period beginning after the extrudate exits the die and extending to before the membrane is wound onto a core or reel, is important to attain the desired permeability of the membrane. During this time period, for a given composition, the permeability of the membrane is determined largely by the cooling rate to which the extrudate is subjected. Permeability is increased by rapid quenching of the extrudate, compared to the permeability obtained from a less drastic quench or slower gelling of the extrudate. Increasing permeability of the membranes, which results from more rapid quenching, normally affects the ability of the membranes to transport water, or other liquids and compounds across the thickness dimension of the membranes. Thus, the extrudate cooling rate (as affected by the temperature and composition of the cooling medium employed) may be varied to modify the permeability of the resulting membrane.

Detailed Description Text (22):

In one method according to the present invention, a polysulfone hollow-fiber extrudate is quenched in air. Within the quench zone, the hollow fibers gel and solidify. The temperature of the air-quench zone is preferably less than about 27.degree. C., more preferably less than about 24.degree. C. The hollow fibers are held in the air zone for preferably less than about 180 minutes, more preferably less than about 30 minutes.

Detailed Description Text (23):

In another preferred method according to the present invention, the hollow-fiber extrudate is quenched in a liquid that is substantially a non-solvent for the polysulfone compound, such as water, poly(ethylene glycol), di(ethylene glycol), tri(ethylene glycol), glycerol, or a mixture thereof. A mixture of solvent(s) and non-solvent(s) alternatively may be used so long as the mixture remains substantially a non-solvent for the polysulfone compound. When a liquid quench comprises water and one or more other components, the ratio of water to the other components is preferably from about 0.25:1 to about 200:1. The temperature of the liquid quench zone is preferably less than about 50.degree. C., more preferably less than about 25.degree. C., and more preferably less than about 10.degree. C. The advantage of a liquid quench is that it offers less resistance to the transfer of heat from the extrudate to the cooling medium than is present in an air quench and, thus, results in a more rapid removal of heat from the extrudate as the membrane forms. The rapid removal of heat modifies the permeability of the resulting membrane and can be used to tailor membrane permeability for the intended

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end use.

Detailed Description Text (25):

In another preferred method according to the present invention, as illustrated in FIG. 2, following quenching, the membrane is passed through at least one leach bath containing a liquid that is substantially a non-solvent for the polysulfone compound, such as water or a mixture of water and sulfolane and/or the non-solvent compound, such as water or a mixture of water and the solvent utilized in the melt composition. Good results have been achieved when the leach bath is water. The membrane is leached to remove at least a portion of the solvent and the non-solvent. The leach bath need not remove all of the solvent and non-solvent from the membrane, depending, at least in part, on the anticipated end use of the membrane.

Detailed Description Text (26):

The minimum temperature of the leach bath is such that removal of the solvent and non-solvent from the membrane occurs at a reasonable rate relative to production rate demands. The minimum temperature of the leach bath is preferably at least about 20.degree. C., more preferably at least about 40.degree. C. The maximum temperature of the leach bath is below a temperature at which the integrity of the membrane is deleteriously affected. Accordingly, the temperature of the leach bath is preferably less than about 95.degree. C.

Detailed Description Text (27):

By way of example, the residence time of a hollow-fiber membrane in the leach bath is preferably less than about 1200 seconds, more preferably less than about 300 seconds. The hollow fiber may, optionally, be drawn to the desired size prior to entrance into the leach bath, during the residence time in the leach bath, subsequent to exiting the leach bath, or during any combination thereof.

Detailed Description Text (28):

Following immersion in the leach bath, the membrane may, optionally, be passed through a rinse bath containing water. The rinse bath removes residues in the membrane from the leach process. The rinse bath is preferably maintained at room temperature. For a hollow fiber, the residence time of the fiber within the rinse bath is preferably less than 1200 seconds, more preferably less than 300 seconds.

Detailed Description Text (29):

After leaching, the membrane may then be subjected to a replasticization process. For hollow-fiber membranes to be used for dialysis, a replasticization bath is used that preferably contains less than about 50 weight percent glycerol and more preferably less than about 45 weight percent glycerol, with the balance being water. The minimum temperature of the replasticization bath is such that replasticization of the membrane occurs at reasonable rate relative to production demands. For example, the minimum temperature of a glycerol-containing replasticization bath is preferably at least about 20.degree. C., more preferably at least about 35.degree. C. The maximum temperature of the replasticization bath is below a temperature at which the membrane integrity could be adversely affected. Accordingly, the maximum temperature of the replasticization bath is preferably less than about 100.degree. C., more preferably less than about 50.degree. C.

Detailed Description Text (30):

Following removal of the membrane from the replasticization bath, excess liquid adhering to the membrane may optionally be removed, preferably by use of a conventional air knife operating at a pressure of about 10 psig to about 60 psig. With hollow fibers, good results have been achieved when the air knife is maintained at a pressure of about 30 psig.

Detailed Description Text (31):

The resulting polysulfone membrane may, optionally, be dried in an oven (preferably a convection oven). The oven is maintained at a temperature of from about

20.degree. C. to about 200.degree. C. With hollow fibers, good results have been achieved when the temperature of the oven is about 70.degree. C. In a convection oven the membrane is dried for a period of from about 5 seconds to about 1200 seconds. With hollow fibers, good results have been achieved when the fiber was dried for at least about 140 seconds.

Detailed Description Text (32):

The semipermeable polysulfone membranes formed by the described methods may be used in liquid-separation processes such as, but not limited to, microfiltration, ultrafiltration, dialysis, and reverse osmosis. The specific fabrication method that is employed, within the scope of methods according to the present invention, is selected so as to tailor the resulting membrane for its anticipated end use. Such adaption is readily achieved by one skilled in the art based upon the teachings herein.

Detailed Description Text (35):

A composition was prepared comprising about 36 weight percent Udel P1835NT11, a brand of bisphenol A polysulfone (available from Amoco Polymers, Inc. of Alpharetta, Ga.) about 44.3 weight percent anhydrous sulfolane (available from Phillips Chemical Company of Borger, Tex.) and about 17.7 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (available from Dow Chemical Company of Midland, Mich.). The solvent to non-solvent ratio was about 4.5:1. The composition was compounded in a co-rotating twin-screw extruder at about 132.degree. C. The extruded composition was cooled, pelletized using an RCP 2.0 pelletizer (available from Randcastle Extrusion Systems, Inc., of Cedar Grove, N.J.), and then remelted and extruded through a 30-hole hollow-fiber spinneret at about 149.degree. C. using a single-screw extruder. The resulting hollow-fiber extrudate was quenched in air at about 21.degree. C. for about 15 seconds, drawn from a first godet (rotating at a surface speed of 172 feet per minute) to a second godet (rotating at a surface speed of 182 feet per minute) to increase the fiber's length by about 5.75 percent, wound on a core, and soaked in a water bath at a temperature of about 25.degree. C. for about four hours.

Detailed Description Text (38):

where K._{sub.b} is the resistance to mass transfer within the fluid present in the lumen of the hollow fiber, and P._{sub.m} is the membrane permeability. It was not possible to determine the membrane permeability, P._{sub.m}, alone using the test apparatus because the flow of solution through an individual fiber could not be made large enough to render K._{sub.b} negligible.

Detailed Description Text (39):

This hollow-fiber membrane could be fabricated into a suitable device for use as an ultrafiltration cell for the removal of contaminants from water or aqueous solutions.

Detailed Description Text (41):

A composition was prepared comprising about 36 weight percent Udel P1835NT11 polysulfone (Amoco Polymers, Inc.), about 45.7 weight percent anhydrous sulfolane (Phillips Chemical), and about 18.3 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical), yielding a solvent to non-solvent ratio about 2.5:1. The composition was compounded in a co-rotating, twin-screw extruder at about 173.degree. C. The extruded composition was then pelletized, remelted, and extruded through a 30-hole hollow-fiber spinneret at about 178.degree. C. using a single-screw extruder. The resulting hollow-fiber extrudate was quenched in air at about 22.degree. C. for 7-8 seconds. The resulting hollow-fiber membrane was wound on a core at about 110 feet per minute, and held dry for about one hour. The hollow fiber was then placed in a water bath maintained at a temperature of about 25.degree. C. for a period of about 12-15 hours.

Detailed Description Text (43):

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The resulting hollow fiber had an average lumen diameter of about 142 .mu.m, and an average wall thickness of about 31 .mu.m. Dialysis test units each containing 150 of the hollow fibers were fabricated. The average in vitro water flux of these devices was $68.0 \text{ mL}/(\text{hr}.\cdot\text{multidot.mmHg}.\cdot\text{multidot.m}.\cdot\text{sup.2})$ and the average K.sub.ov for sodium chloride was about $2.28 \cdot 10^{-2}$ centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber. This hollow-fiber membrane is useful for ultrafiltration, such as for use in an ultrafiltration cell for the removal of contaminants from water or aqueous solutions.

Detailed Description Text (45):

A composition was prepared comprising about 38 weight percent Udel P1835NT11 polysulfone (Amoco Polymers, Inc.), about 44.3 weight percent anhydrous sulfolane (Phillips Chemical), and about 17.7 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical), yielding a solvent to non-solvent ratio of about 4.5:1. The composition was compounded in a co-rotating, twin-screw extruder at about 99.degree. C., and extruded directly through a 30-hole hollow-fiber spinneret. The extrudate was quenched in air at about 26.degree. C. for about 6 seconds. The resulting hollow-fiber membrane was wound on a core at about 160 feet per minute, and placed immediately into a water bath for a period of about 12-15 hours.

Detailed Description Text (47):

The resulting hollow-fiber membrane had an average lumen diameter of 237 .mu.m, and an average wall thickness of 35 .mu.m. Dialysis test units each containing 150 of the resulting fibers were fabricated from this fiber. The average in vitro water flux of these devices was $143.5 \text{ mL}/(\text{hr}.\cdot\text{multidot.mmHg}.\cdot\text{multidot.m}.\cdot\text{sup.2})$ and the average K.sub.ov for sodium chloride was found to be $0.88 \cdot 10^{-2}$ centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber. This hollow-fiber membrane can be used in an ultrafiltration cell for the removal of contaminants from water or aqueous solutions.

Detailed Description Text (49):

A composition was prepared comprising about 38 weight percent Udel P1835NT11 polysulfone (Amoco Polymers, Inc.), about 45.7 weight percent anhydrous sulfolane (Phillips Chemical), and about 18.3 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical), yielding a solvent to non-solvent ratio of about 2.5:1. The composition was compounded in a co-rotating twin-screw extruder at about 143.degree. C., and extruded directly through a 30-hole hollow-fiber spinneret. The extrudate was quenched in air at about 25.degree. C. for about 0.08 minutes, wound on a core at about 203 feet per minute, and held dry for thirty minutes before being placed in a 25.degree. C. water bath for about three days.

Detailed Description Text (51):

The resulting hollow-fiber membrane had an average lumen diameter of 192 .mu.m, and an average wall thickness of 29.5 .mu.m. Dialysis test units each containing 150 of the resulting fibers were fabricated. The average in vitro water flux of these devices was $141.2 \text{ mL}/(\text{hr}.\cdot\text{multidot.mmHg}.\cdot\text{multidot.m}.\cdot\text{sup.2})$ and the average K.sub.ov for sodium chloride was found to be $1.20 \cdot 10^{-2}$ centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber. This hollow-fiber membrane is useful in an ultrafiltration cell for the removal of contaminants from water or aqueous solutions.

Detailed Description Text (53):

A composition was prepared comprising about 34 weight percent Udel P1835NT11 polysulfone (Amoco Polymers, Inc.), about 54 weight percent anhydrous sulfolane (Phillips Chemical Company), about 11 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical Company), and about

1 weight percent glycerol (VanWaters & Rogers, Inc., Seattle, Wash.), yielding a solvent to non-solvent ratio of about 4.5:1. The composition was compounded in a co-rotating twin-screw extruder at about 144.degree. C. The extrudate was then cooled, pelletized, remelted, and extruded through a 30-hole hollow-fiber spinneret at about 134.degree. C. using a single-screw extruder. The resulting extrudate was quenched in air at about 20.degree. C. for about 0.08 minute, and wound on a core at about 200 feet per minute. The entire wound core was immediately placed in a 25.degree. C. water bath for a period of about 15-20 hours.

Detailed Description Text (55):

The resulting hollow-fiber membrane had an average lumen diameter of about 165 .mu.m, and an average wall thickness of about 18 .mu.m. Test units each containing about 150 of the resulting fibers were fabricated. The average in vitro water flux of these devices was 67.2 mL/(hr mmHg m.sup.2) and the average K.sub.ov for sodium chloride was found to be 2.19.times.10.sup.-2 centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber.

Detailed Description Text (57):

A composition was prepared comprising about 34 weight percent Udel A P1835NT11 polysulfone (Amoco Polymers, Inc.), about 54 weight percent anhydrous sulfolane (Phillips Chemical Company), about 6 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical Company) and about 6 weight percent tri(ethylene glycol) (Aldrich Chemical Company, Inc., Milwaukee, Wis.), yielding a solvent to non-solvent ratio of about 4.5:1. The composition was compounded in a co-rotating, twin-screw extruder at about 153.degree. C. The extrudate was then cooled, pelletized, remelted, and extruded through a 30-hole hollow-fiber spinneret at about 137.degree. C. using a single-screw extruder. The resulting hollow-fiber extrudate was quenched in air at about 20.degree. C. for 0.08 minute, and wound on a core at about 200 feet per minute. The entire fiber core was immediately placed in a 25.degree. C. water bath for a period of 15-20 hours.

Detailed Description Text (59):

The resulting hollow-fiber membrane had an average lumen diameter of about 180 .mu.m, and an average wall thickness of about 20 .mu.m. Test units each containing about 150 of the resulting fibers were fabricated. The average in vitro water flux of these devices was 60.0 mL/(hr.multidot.mmHg.multidot.m.sup.2) and the average K.sub.ov for sodium chloride was found to be 2.17.times.10.sup.-2 centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber.

Detailed Description Text (61):

A composition was prepared comprising about 32 weight percent Udel P1835NT11 polysulfone (Amoco Polymers, Inc.), about 53 weight percent anhydrous sulfolane (Phillips Chemical Company), and about 15 weight percent poly(ethylene glycol) having an average molecular weight of about 1000 daltons (Dow Chemical Company), yielding a solvent to non-solvent ratio of about 2.5:1. The composition was compounded in a co-rotating twin-screw extruder at about 131.degree. C. and extruded directly through a 30hole hollow-fiber spinneret. The extrudate was quenched in water at about 7.degree. C. for about 6 seconds. The resulting hollow-fiber membranes were wound on a core at about 244 feet per minute. The entire fiber core was immediately placed in a 25.degree. C. water bath for a period of about 15-20 hours.

Detailed Description Text (63):

The resulting hollow-fiber membranes had an average lumen diameter of about 203 .mu.m, and an average wall thickness of about 37 .mu.m. Test units each containing about 150 of the resulting fibers were fabricated. The average in vitro water flux of these devices was 9.1 mL/(hr.multidot.mmHg.multidot.m.sup.2) and the

average K.sub.ov for sodium chloride was found to be 1.76.times.10.sup.-2 centimeters per minute at a solution flow rate through the fiber lumina of about 0.02 milliliters per minute per fiber.

Other Reference Publication (2):

Cabasso et al., "Polysulfone Hollow Fibers. I. Spinning and Properties," J. Appl. Polymer Sci. 20:2377-2394 (1976).

Other Reference Publication (3):

Cabasso et al., "Polysulfone Hollow Fibers. II. Morphology," J. Appl. Polymer Sci. 21:165-180 (1977).

Other Reference Publication (4):

Hu et al., "Preparation of Polysulfone (PSF) Thermal Phase Inversion Membrane," Chem. Abstracts, vol. 123, No. 6, Aug. 7, 1995.

CLAIMS:

1. A melt spinning process for making a polysulfone semipermeable membrane, the process comprising the steps of:

(a) forming a composition including a polysulfone compound, a solvent for the polysulfone compound, and a non-solvent for the polysulfone compound, the solvent and non-solvent being present in the composition in a ratio sufficient to form a semipermeable membrane useful for a liquid separation process;

(b) heating the composition to a temperature at which the composition is a homogeneous liquid;

(c) extruding the homogeneous liquid to form an extrudate; and

(d) thermal quenching the extrudate to cause a phase separation and thereby form a semipermeable membrane.

3. The process of claim 1, wherein polysulfone compound is a polyarylsulfone compound.

4. The process of claim 1, wherein the polysulfone compound is selected from the group consisting of bisphenol A polysulfone, polyether polysulfone, polyphenyl polysulfone, and mixtures thereof.

7. The process of claim 1, further comprising the step of drawing the semipermeable membrane.

8. The process of claim 1, further comprising the step of storing the semipermeable membrane in a liquid for a period of about 4 hours to about 15 days.

9. The process of claim 8, further comprising the step of passing the semipermeable membrane through a leach bath.

10. The process of claim 8, wherein the leach bath comprises a non-solvent for the polysulfone compound.

12. The process of claim 9, further comprising the step of passing the semipermeable membrane through a rinse bath.

14. The process of claim 12, further comprising the step of passing the semipermeable membrane through a replasticization bath.

17. The process of claim 14, further comprising the step of drying the

semipermeable membrane.

18. The process of claim 17, wherein the semipermeable membrane is dried in an oven.

19. A melt-spinning process for making a polysulfone semipermeable membrane useful for a liquid membrane separation process, the process comprising the steps of:

(a) forming a composition including a polysulfone compound, a solvent for the polysulfone compound, and a non-solvent for the polysulfone compound, wherein the solvent and non-solvent are present in a ratio sufficient to form a semipermeable membrane useful for a liquid separation process;

(b) heating the composition to a temperature at which the composition is a homogeneous liquid;

(c) forcing the homogeneous liquid through a strand die to produce solid strands;

(d) thermal quenching the homogeneous liquid to produce strands;

(e) pelletizing the strands to a particle size readily fed to an extruder;

(f) remelting the particles to form a liquid; and

(g) extruding the liquid to form an extrudate and thermal quenching the extrudate to cause a phase separation and form a semipermeable membrane.

22. The process of claim 19, wherein the polysulfone compound is a polyarylsulfone compound.

24. The process of claim 19, further comprising the step of drawing the semipermeable membrane.

25. The process of claim 19, further comprising the step of storing the semipermeable membrane in a liquid for a period of about 4 hours to about 15 days.

26. The process of claim 19, further comprising the step of passing the semipermeable membrane through a leach bath.

27. The process of claim 26, wherein the leach bath comprises a non-solvent for the polysulfone compound.

29. The process of claim 19, further comprising the step of passing the semipermeable membrane through a rinse bath.

31. The process of claim 19, further comprising the step of passing the semipermeable membrane through a replasticization bath.

34. The process of claim 19, further comprising the step of drying the semipermeable membrane.

35. The process of claim 34, wherein the semipermeable membrane is dried in an oven.